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The work under this contract was aimed at developing a better understanding of the preparation parameters and microstructural features that control magnetic hardness in alloys of the type $Sm_2(Co,Fe,Cu,T)_{17}$. (Here, T is one or more additional transition metal.) The basic intermetallic compound Sm_2Co_{17} and its modifications with Fe have very high uniaxial crystal anisotropy, which promises the possibility of good permanent magnets, but ingots, powders, or sintered bodies were found to always have low coercive force. Research workers at the TDK, Hitachi, and other

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20. ABSTRACT CONTINUED

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A STUDY OF THE MAGNETIC AND METALLURGICAL
PROPERTIES OF Sm (Co, Fe, Cu, Zr)$_z$ ALLOYS
WITH Z = 8.5

FINAL SUMMARY REPORT

Dr. Alden E. Ray
Dr. Karl J. Strnat

28 JANUARY 1981

U.S. ARMY RESEARCH OFFICE

CONTRACT NO. DAAG29-79-C-0150

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A STUDY OF THE MAGNETIC AND METALLURGICAL
PROPERTIES OF $Sm(Co,Fe,Cu,Zr)_z$
ALLOYS WITH Z=8.5

FINAL SUMMARY REPORT

by

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January 28, 1981

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A. FOREWORD AND PROBLEM STATEMENT

The work under this contract was aimed at developing a
better understanding of the preparation parameters and micro-
structural features that control magnetic hardness in alloys of
the type $Sm_2(Co,Fe,Cu,T)_{17}$. (Here, T is one or more additional
transition metal.) The basic intermetallic compound Sm_2Co_{17}
and its modifications with Fe have very high uniaxial crystal
anisotropy, which promises the possibility of good permanent
magnets, but ingots, powders, or sintered bodies were found
to always have low coercive force.

Research workers at the TDK, Hitachi, and other com-
mercial laboratories in Japan reported that by adding small
percentages of copper plus one other metal of the group Zr,
Hf, Ti, or Mn, and by fairly complicated heat treatments, it is
possible to produce usefully high coercive forces in the 5-7 kOe
regime. Quite recently, coercivities twice as high were reported
by TDK. These companies have begun producing commercial
permanent magnets based on such alloys, while no U.S. firm
or laboratory has yet succeeded in even approaching these
results. American users must buy such magnets abroad.

We undertook it to try and duplicate the reported alloys
and procedures, using the TDK work with T=Zr as the model. An
understanding of the relationship between basic magnetic
properties, microstructure, and coercivity is to be developed
by our efforts and those of cooperating laboratories. And
finally, we hope to go on toward formulating compositions,
preparation procedures, and heat treatments that can lead to
the development of better magnets. We set out to do all phases
of the necessary work initially in the University of Dayton

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laboratories: From the alloy preparation in kilogram quantities, annealing and metallographic/structural characterization, through the measurement of magnetic alloy properties, and through the powder-metallurgical steps of making sintered magnet bodies, to precipitation annealing experiments to optimize coercivity and hysteresis loop shape by means of developing the proper microstructures.

Although our proposal had ambitiously promised studies on Mn and Cr-containing new alloys, it turned out that the mere task of repeating the preparative and powder-metallurgical developments (that had taken five years or so of intense efforts in the Japanese laboratories) was quite formidable and required all of the available time and funds. We can now report that through the efforts under this contract (and complementary work with industrial sponsorship that was more directly aimed at making sintered magnets) we have essentially mastered the arts of preparing REC 30-like alloys in the proper metallurgical state, sintering them to high density, and developing high coercive forces by proper post-sintering heat treatments. The more basic science aspects of relating the coercivity and domain-wall motion to anisotropy and microstructure have so far been neglected. However, it was absolutely necessary first to create the materials and magnets in their proper states and to characterize them in terms of their overall properties, before the sub-microscopic structure studies that seem to be needed for a true understanding make any sense. Similarly, it was necessary to "reinvent" the proper complex sequence of preparation steps, using a proven alloy as a vehicle, before attempts to improve matters by compositional variations have a reasonable chance of success.

This report first describes the alloy preparation effort which had to precede any magnetic characterization or magnet preparation work. In the second half, we report on the results of measurements of basic magnetic properties of the alloys, powder preparation, field pressing, and sintering experiments.

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B. PREPARATION OF THE ALLOYS

1. INTRODUCTION

This investigation required the preparation of 1 kg
quantities of an alloy corresponding to the composition of
type REC-30 magnets produced by the TDK Electronics Corporation.
The composition of REC-30 magnets is reported to be, by weight,
25.5% Sm, 50% Co, 15% Fe, 8% Cu, and 1.5 % Zr. Several
problems are associated with the preparation of an alloy of
this type to close compositional tolerances, primarily due to
the relatively high reactivities of the Sm and Zr components
and the lack of commercially available melting crucibles which
are inert to both the rare earth and transition metal components.
We have employed somewhat modified melting procedures developed
by us for the preparation of similar quantities of binary,
RCo_5-type alloys to successfully prepare the required alloys.

2. EXPERIMENTAL PROCEDURES

Alloys were prepared by induction melting the constituent
elements followed by chill casting. Melting and casting was
done in a puried argon atmosphere at 0.8-0.9 Torr. Power to
the melting crucible was supplied by a 15 KW, 9600 Hz motor
generator.

The melting crucibles were right cylinders, 3.5" O.D. by
6" high, made of 99% pure Al_2O_3 with an apparent porosity of
19%. Melt temperature was measured and controlled by means
of a high density alumina sheathed, Pt-Pt, 13 Rh thermocouple
located at the bottom of the melting crucible. The molten alloys
were cast into a flat, 6" square by 0.75" deep, water-cooled
copped mold.

An alloying additions assembly is located above the melting
crucible. This assembly consists of two 1-3/8" I.D. stainless

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steel tubes containing pistons connected to 1/4" bolts which protrude through slots at the top of the tubes. The tubes are attached to a mechanical feedthrough at the top of the melting chamber. A hooked rod attached to a second feedthrough serves to pull the piston forward so that the contents of the tubes can be added to the alumina melting crucible. A viewing port is located directly above the melting crucible so that the tubes and hooked rod can be visually positioned and the alloying additions made in a controlled manner. The capacity of the alloying addition tubes is approximately 2.0 kg of arc melted cobalt buttons. Each tube will hold 18 to 20 1-1/4" diameter cobalt buttons weighing approximately 50 grams each.

For the first attempt to prepare the TDK REC-30 alloy, the melting crucible was charged with all of the samarium (255 gm) and sufficient cobalt (65 gm) to produce a near eutectic mixture of Sm-Co (20 w/o Co). The alloy additions hopper was charged with approximately 50 gm arc melted buttons containing the balance of the cobalt and the other transition metals. The Zr (15 gm) and the remaining Co (435 gm) were arc melted together to make 9 buttons (with a total weight, after arc melting, of 449.2 gm). The purpose of prealloying the Zr with the Co was to ensure adequate mixing of the Zr in the melt. Also a 15 gm button is a little too small to feed conveniently from the alloying additions hopper.

In order to minimize reaction of the alloy with the alumina melting crucible, the alloy was heated as rapidly as possible, within the thermal shock limits of the crucible. Also the melt temperature was maintained just above the liquidus of the melt composition, which increases incrementally as the transition metal buttons are added.

The degree of reaction between the melt and alumina crucible increases rapidly with temperature, so that the holding time at the pouring temperature should be just long enough to allow complete melting and homogenization of the melt after the final alloying addition of cobalt.

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A button drop vs. temperature schedule was prepared by estimating the approximate liquidus of the melt as a function of the number transition metal buttons added from the alloying additions tubes, and the desired heating rate. The first melt was poured at a temperature of 1375°C, 86 minutes after heating was initiated, and three minutes after the last transition metal button was dropped into the melt.

Chemical analysis showed no detectable zirconium in the cast alloy of the first melt. Apparently zirconium reacts with, and is absorbed by the alumina crucible. The second attempt to prepare the REC-30 alloy differed from the first in that all of the zirconium was contained in a single cobalt button, and this button was the 14th and last button dropped into the melt. Chemical analysis of the second melt showed a substantial amount of the zirconium was retained in the cast alloy. Approximately 5 w/o and 7 w/o of the samarium were lost in the first melt and second melt, respectively. To compensate for the seemingly unavoidable samarium loss, the nominal samarium content of the third melt was increased by 10%, and this additional samarium was added last, simultaneously with the zirconium addition.

A fairly rapid, but smooth pouring rate resulted in very little splatter. When the surface of the cast alloys cooled to a cherry red color (800-900°C) large cracks (accompanied by audible popping noise) were observed. Similar behavior was observed for cast RCo_5-type alloys when they reached a dull red color (700-800°C). Since these temperatures are in the range of the Curie temperatures of the predominant alloy phases present, it is believed the breaking up of the alloy casting results from anisotropic dimensional changes as the phases cool through their magnetic ordering temperatures.

3. RESULTS

The first two melts of simulated REC-30 alloys were identical in nominal composition, weighing 1000 gm and containing

255 gm Sm, 500 gm Co, 150 gm Fe, 80 gm Cu, and 15 gm Zr. The third melt contained 280 gm Sm for a total initial weight of 1025 gm. The main portions of the cast alloys were recovered as broken pieces, approximately 6 mm thick. Small amounts of each alloy were lost by splatter during casting, as tiny fragments formed when the alloys broke up while cooling through their magnetic ordering temperatures, and by vaporization losses. The major losses in each case were due to alloy adhering to the crucible walls and by reaction of the molten alloys with the crucibles. The actual yields of the three melts are given in Table I. Samples were taken from the front, middle, and back of each casting and sent to the Bowser-Morner Testing Laboratories, Dayton, Ohio for chemical analysis by X-ray fluorescence spectroscopy. The results are given in Table II.

The microstructures of the cast alloy slabs were virtually identical. Fine-grained chill layers, 0.5 to 1.0 mm thick, were formed by the first molten alloy contacting the water-cooled copper mold. Above the chill layers, the microstructures were relatively uniform, consisting of about 70% of an apparently congruently solidifying phase surrounded by a eutectic-like matrix. The grain size of the high temperature phase averaged about 30 µm but ranged from 5 µm to 80 µm. Although the two layers in the cast slabs were metallographically distinct, homogenization of the cast alloys for 2 hours at 1150-1175°C produced essentially uniform, nearly single-phased microstructures, indicating compositional uniformity between the layers.

4. CONCLUSIONS

The melting and casting procedures employed were successful in producing a 1 kg alloy close to the reported composition of REC-30 magnets. The very high degree of reactivity between the zircomiun component and the alumina melting crucibles was not anticipated. The zirconium loss was minimized by adding this component last and pouring shortly after its addition to the melt.

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TABLE I

YIELDS OBTAINED FOR THREE MELTS OF
SIMULATED REC-30 ALLOY

	1st melt	2nd melt	3rd melt
Cast alloy, large pieces	920 gm (92.0%)	917 gm (91.7%)	937 gm (91.4%)
Fragments, splatter (recovered)	10 gm (1.0%)	6 gm (0.6%)	6 gm (0.6%)
Weight grain of crucible	68 gm (6.8%)	75 gm (7.5%)	80 gm (8.0%)
Unrecovered (by difference)	2 gm (0.2%)	2 gm (0.2%)	2 gm (0.2%)

TABLE II

CHEMICAL ANALYSES OF THE SIMULATED REC-30 ALLOY
CASTINGS (IN WEIGHT %, AND SCALED TO 100)

1st Melt

Element	Front	Middle	Back	Average
Sm	24.33	24.17	24.09	24.20
Co	51.84	51.95	52.02	51.94
Fe	15.61	15.64	15.72	15.66
Cu	8.21	8.23	8.17	8.20
Zr	<.04	<.04	<.04	<.04

2nd Melt

Element	Front	Middle	Back	Average
Sm	23.66	23.92	23.65	23.74
Co	52.20	51.90	52.18	52.09
Fe	15.16	15.12	15.20	15.16
Cu	8.04	8.15	8.03	8.07
Zr	0.93	0.92	0.93	0.93

3rd Melt

Element	Front	Middle	Back	Average
Sm	25.20	25.37	25.32	25.30
Co	50.97	50.69	50.74	50.80
Fe	15.08	15.10	15.06	15.08
Cu	7.91	8.00	8.05	7.99
Zr	0.84	0.84	0.84	0.84

Some samarium loss by reaction with the crucible and by vaporization was anticipated. Samarium loss by vaporization proved minimal, and its loss by crucible reaction, while significant, appears predictable and can be compensated by increasing the amount of samarium added above that required in the final alloy. The experience gained in these experiments suggest that both samarium and zirconium losses would be reduced by melting the cobalt, iron, and copper components first, and adding all the samarium and zirconium constituents last. The alloy should then be held at temperature just long enough to ensure complete mixing prior to pouring.

C. BASIC MAGNETIC CHARACTERIZATION

1. INTRODUCTION

For any alloy to be used in permanent magnets of the R-Co type, we need to know at least the following bulk properties: the saturation magnetization at room temperature, M_S; the curie temperature, T_C; and the crystal anisotropy, usually characterized by the anisotropy field, H_A. The saturation defines an upper limit for the useful remanent flux density of a magnet made from the alloy, $B_r \leq B_S = 4\pi M_S$ [Gauss], and it also sets a limit on the theoretically possible energy product, $(BH)_{max} \leq 4\pi^2 M_S^2$ [MGOe]. The curie point relates to the temperature coefficient of the magnet's remanence and the upper limit on the use temperature. A high anisotropy field is a precondition for a high intrinsic coercive force, although $_M H_C$ is influenced by many parameters in a way as yet poorly understood.

Because of this, we measured these three quantities on all alloys prepared under this program, and also on sintered magnet samples obtained from the TDK Research Laboratory.

2. PROCEDURES

(a) The "saturation" magnetization is determined by measuring an M versus H curve to $\sim H=25$ kOe with a vibrating sample or oscillating specimen magnetometer. The sample is a powder of <37 µm particles which consists mostly of single crystals. The particles are free to rotate and align with the field. A good approximation of σ_S is easily obtained from the highest moment value measured, and pycnometric determination of the density, ρ, permits calculation of M_S or B_S.

(b) The anisotropy field, H_A, is also obtained from magnetometric σ versus H curves. These are measured on fine

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powder samples that are first oriented in a magnetic field and "frozen" in an epoxy-resin matrix. Measurements with the field parallel and perpendicular to the alignment axis permit a good estimate of H_A provided the particle is fairly single-crystalline and the magnetic symmetry shows a single easy axis. Our powders fulfilled these conditions moderately well.

(c) To obtain the curie temperature, we perform a "thermomagnetic analysis" (TMA) by measuring the low-field (1 Oe) permeability at 5 kHz as a function of temperature on an alloy powder. This method also yields information on the phase composition of samples containing several ferromagnetic phases (such as our magnet alloys) and on any structural transitions as a function of temperature which affect the permeability.

3. SUMMARY OF RESULTS

Measurements of this kind were performed on the three alloys prepared, in the as-cast and homogenized states. The ranges of values found were: σ_s = 102.2 to 108.6 emu/g. B_s = 10.7 to 11.4 kG, H_A = 86 to 98.5 kOe, T_c = 760° to 780°C. The saturation values make it appear possible that magnets approaching 29-32 MGOe might be prepared from these alloys.

The equivalent values we measured on reground REC-30 magnets from TDK, our reference material, were σ_s = 104, B_s = 10.96 kG, H_A = 96 kOe, T_c = 780°C. The TMA of both the REC-30 and our alloy gave indications of a partial (eutectoid?) decomposition of the 2-17 phase in the temperature range 500°-800°C.

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D.   POWDER METALLURGICAL MAGNET PREPARATION

1.   INTRODUCTION

To prepare a permanent magnet from a 2-17 alloy, it is necessary to pulverize the ingot to a particle size range of 3-8 μm, compact the powder to a density of 60-70% by compaction in a pressing die that permits the application of a magnetizing and aligning field, to outgas and sinter the compact to a density between 90 and 100%, and then to subject it to the proper sequence of annealing and cooling steps.   The samples so produced must be characterized at different steps of the preparation process.   Dimensions, density, and magnetic hysteresis loops must be measured, the micro-structure metallographically studied, and the results must be correlated with details of the processing parameters.

2.   TOOLS AND PROCEDURES

(a) Powder Properties: Steel and porcelain mortars, together with Tyler screens, are used to prepare defined feed fractions for the ball mill.   The fine powders are prepared in an attritor mill, grinding with stainless steel balls in toluene under a dry N_2 gas blanket.   The particle size is measured with a Fisher subsieve-sizer (after drying) and occasionally is checked with the help of a scanning electron microscope .

(b) Pressing: We used two sample types in these investiga-tions, cylindrical compacts of 1/2" diameter pressed with force and aligning field (8 kOe) parallel, and bricks (1/4" x 1/4" x 3/4") or cubes (1/4") pressed with a transverse aligning field up to 36 kOe.   For best results, high-coercivity powders had to be premagnetized in a 100 kOe pulsed-field magnetizer.

(c) Sintering: For this we use a dual-barrel inconel tube furnace capable of 1300°C and 10^{-7} Torr vacuum.   Each half of

the furnace can be independently temperature programmed, pumped or filled with gas. We normally backfill the tube after out-gassing with highly purified argon as the atmosphere for sintering and heat treating. The samples, on a tray, can be withdrawn from the hot zone to a quench plate for rapid cooling. The temperature/time profile is recorded from a thermocouple in contact with the inconel sample tray.

(d) Density Measurement: Magnets, lumps of the alloy, or coarse powders are weighed in air and iso-octane to determine the mass density by pycnometry. Weighing bottles or suspended samples are used, as appropriate. Relative densities of sintered bodies (and volume shrinkage) are calculated by comparing the magnet density with that of the homogenized bulk alloy. Pycnometric rather than x-ray densities must be used because the alloys and magnets typically contain several phases.

(e) Magnetic Characterization: Magnetization curves (virgin and loops) are measured with a hysteresigraph that employs inductive B and H-signal acquisition with coils near the sample surface, electronic integration and (B-H) compensation. Intrinsic loops and second-quadrant demagnetization curves are plotted. B versus H curves and energy products are computed from these.

(f) Post-Sintering Heat Treatments: The samples are usually subjected to a solutionizing heat treatment right after sintering, then quenched, removed from the furnace tube and measured (density, hysteresis loop). Then they are restored to the furnace and given the prolonged heat-treating sequence necessary to develop high coercive force and a good second-quadrant hysteresis loop shape. This is done in the second barrel of the same furnace. In all experiments for this project, an argon atmosphere was used.

3.   EXPERIMENTS AND RESULTS

(a) Grinding Experiments: First we had to become familiar with the grinding/ball milling behavior of these new alloys. Two attritor ball-mill runs were done on REC-30 sintered magnet scrap and on the as-cast, Zr-free alloy. For learning purposes, samples were taken during the milling in order to monitor the rise of the coercive force. Next, 50g charges of all four alloys (REC-30, homogenized Zr-free, low-Zr and high-Zr melts made by us were milled to what we thought to be the optimum particle size for pressing and sintering.

(b) Pressing Samples for Sintering: The powders were then compacted in a 1/2" cylindrical die with field and force parallel to each other. Attempts to press with a transverse aligning field failed to yield integral compacts. This was attributed in part to the fact that Be-Cu was too soft a die-wall material, and these hard powder particles were pressed into the die, causing severe stresses in the sample during removal. Lining the die with WC and lowering the compresion pressure to about 35 kpsi has overcome these problems, so we can now successfully press in a transverse field (up to 36 kOe).

(c) Early Sintering Experiments: Four different sintering experiments were performed on the powder compacts as produced. The sintered bodies looked good and were well-densified, but had low coercive force (150-250 Oe). This was expected, because at this stage the copper is retained in solid solution during the rapid cooling from the sintering temperature.

(d) Post-Sintering Heat Treatments: Five different heat treatments aimed at developing coercive force were tried, ranging from a "step-treatment" described by TDK, to a "ramp" heat-treatment described by a Chinese group as a supposed improvement and simplification of the TDK procedure. In these, we were not able to push $_M H_c$ higher than 1.3 kOe. In fact, the highest

value obtained up to that date was only 3.9 kOe; this was on a sample cooled moderately slowly after homogenization at 1175°C, alas at an uncontrolled cooling rate that was not measured. It turned out that the "recipes" described in the Japanese and Chinese publications are incomplete and quite vague in important details, so that the "initial" task of trying to reproduce the TDK composition and magnet properties became a lengthy and frustrating project in itself. However, it was imperative that we learn to duplicate the best published results before we can intelligently proceed with sintering work on new alloy compositions or try to modify the post-sintering heat treatment.

(e) Samples for the U.S. Army ET & DL Laboratory: From the sintered samples, 1/8" cubes were machined for the U.S. Army ET & DL for magnetometer measurements and other experiments (in June, 1980). These samples were not good, high-coercivity magnets. They were intended for studying the temperature variation of saturation and crystal anisotropy.

(f) General Remarks: On June 20, 1980, Dr. Leupold of the ET & DL visited the University of Dayton for a detailed review of the work performed. He received samples at that time and a written summary. The contract period was coming to an end then and the funds were exhausted.

We informed the Project Officer then that the University of Dayton had been asked to conduct a related effort for a U.S. industrial firm that has a vital interest in the availability of domestic 2-17 permanent magnets. The company's project has the longer-range objective of developing sintered $RE(TM)_{\sim 7.5}$ magnets in which a substantial portion of the samarium is replaced by other light rare-earth metals, singly or in mixture. The intent is to broaden the raw materials base for a potential large-volume industrial use of such magnets where the heavy dependence on the relatively scarce Sm is perceived as severely limiting. Our effort under that project — while aimed at magnets for a different class of devices and concerned with

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different alloy compositions than our Army-sponsored effort —
would nevertheless share with it our basic requirement, namely,
that we must first master the steps leading to a duplication
of earlier Japanese achievements. While the industrial work
could thus benefit from our earlier (and mostly negative)
experiences with sintering and heat treating, it would also
afford us the opportunity to continue the process of learning
the correct powder-metallurgical procedures. This should in
turn improve our restarting position if and when the ARO should
sponsor the proposed follow-on work to the present project.

The general goal of the Army effort in 2-17 magnets is
to promote the development of high-energy, temperature-
compensated magnets for use in compact microwave devices.
The overriding consideration is high performance. The
availability of raw materials in large quantity at low cost
is less important. In keeping with this, our work statement
calls for increasing the transition metal content toward $x=8.5$
(stoichiometric 2-17) and the use of heavy rare earths (Gd or
Er) for internal temperature compensation. The specific work
was, of course, much more basic. The proposed continuation
effort would emphasize the objective of increasing x over 7.4
$(x \rightarrow 8.5)$ and studying and developing the precipitation mechanisms
that can lead to high coercive force in such alloys.

Recognizing the potential benefits to its overall magnet
R&D program, the University accepted the industrial contract.
In the first half year of that work, we finally succeeded in
making sintered magnets over 20 MGOe energy product from TDK-
like alloys. We have also learned post-sintering heat treat-
ments that yield coercivities of 5-6 kOe for short-time anneals,
10-25 kOe with modified long-term annealing.

E. SUMMARY AND CONCLUSIONS

The work under this ARO contract allowed us to develop reliable procedures with close parameter control for the preparation of magnet alloys in kilogram quantities, homogenization and recrystallization heat treatments, ballmilling, compacting, sintering and post-sinter heat treating. The concrete results — in terms of finding an alloy composition that would yield high-energy magnet samples, or learning the heat treatments necessary for high coercive forces — were poor up to the end of the contract term in the summer of 1980.

Under the above mentioned industrial grant, we have since then succeeded in producing alloys and sintered bodies of very high coercivity and fairly high energy products. In view of this, we feel that we now have the basis to begin work on the modified alloy compositions of particular interest for microwave devices (heavy rare earth substitutions, higher overall transition-metal content) with a good chance of making rapid progress. We are now also in a position to prepare samples in different stages of the heat treatment leading up to good sintered magnets for critical investigations of the relation between microstructure and coercivity.